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MANN TESTING LABORATORIES
REPORT

Reference 26

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82 U6502 V.9

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UPPER OTTAWA LANDFILL SITE

SOIL ANALYSIS

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UPPER OTTAWA LANDFILL SITE

SOIL ANALYSIS

REF. #852660

1.0 INTRODUCTION

Presented in the following report are the results of the analysis for polynuclear aromatic hydrocarbons (PAH's) and polychlorinated biphenyls (PCB's) conducted on a series of soil samples taken from the Upper Ottawa Landfill Site. The PAH analysis was also conducted on material obtained from Dofasco Inc. which has been used as a covering fill at the landfill site.

2.0 ANALYTICAL RESULTS

The concentrations of the PAH's detected is given in Table II. Results of the PCB analysis for the landfill site are listed in Table III. All values are given in ppm (ug/gr) units and have been corrected for both % recovery and sample moisture content.

Analytical standards were not available for the two compounds

- 4-H-cyclopenta-d,e,f-phenanthrene
- 1-H-phenalene

However all the sample chromatograms were screened for their characteristic ions.

TABLE I - Sample Identification

Sample Legend

#	Identification
1	Upper Ottawa Landfill Upstream
2	Upper Ottawa Landfill Downstream
3	Upper Ottawa Landfill Northwest creek by apron
4	Upper Ottawa Landfill Leachate seep
5	Upper Ottawa Landfill South ditch
6	Dofasco Hot Mill (Filtration plant oxides)
7	Dofasco BOF (solidified oxides)
8	Dofasco Steel making (metalic slag fines)
9	Dofasco Cold mill waste (water treatment plant oxides)
10	Dofasco Blast furnace oxides
11	Dofasco Foundary sands

TABLE II UPPER OTTAWA PAH STUDY

CONC(ppm)

COMPOUND	EXTRACT BLANK	SAMPLE #1	SAMPLE #2	SAMPLE #3	SAMPLE #4	SAMPLE #5	MDL (PPM)
BUTYL BENZENE	-	-	-	-	-	-	0.2
NAPHTHALENE	0.14	TR	TR	TR	0.30	TR	0.1
2-METHYL NAPHTHALENE	-	-	-	-	-	-	0.1
DIMETHYL NAPHTHALENE	TR	-	-	-	TR	-	0.2
TRIMETHYL NAPHTHALENE	TR	TR	-	-	-	-	0.1
ACENAPHTHYLENE	-	-	-	-	-	-	0.1
ACENAPHTHENE	-	-	-	-	-	-	0.1
FLUORENE	-	-	-	-	TR	-	0.2
PHENANTHRENE	-	2.02	0.83	0.51	1.05	TR	0.2
METHYL PHENANTHRENE	TR	2.83	10.88	1.71	6.49	13.57	0.2
ANTHRACENE	-	-	-	-	-	-	0.2
METHYL ANTHRACENE	-	-	-	-	-	-	0.4
FLUORANTHENE	-	4.43	1.46	1.04	TR	TR	0.4
PYRENE	-	3.65	1.14	0.97	TR	TR	0.4
BENZO(A)ANTHRACENE	-	-	-	-	-	-	0.4
CHRYSENE	TR	2.13	TR	TR	TR	TR	0.4
BENZO(A)PYRENE	-	-	-	-	-	-	0.6
BENZO(E)PYRENE	-	-	-	-	-	-	0.6
BENZO(B)FLUORANTHENE	-	2.78	1.03	TR	TR	TR	0.6
BENZO(K)FLUORANTHENE	-	-	-	-	-	-	0.6
PERYLENE	-	-	-	-	-	-	0.6
BENZO(GHI)PERYLENE	-	-	-	-	-	-	1.0
INDENO(123-CD)PYRENE	-	TR	TR	TR	-	-	1.0
DIBENZO(AH)ANTHRACENE	-	TR	TR	TR	-	-	1.0
CORONENE	-	TR	-	-	-	-	2.0
1H-PHENALENE	-	-	-	-	-	-	5.0
4H-CYCLOPENTA-D,E,F,PHENANTHRENE	-	-	-	-	-	-	5.0

- NONE DETECTED
MDL = MINIMUM DETECTION LIMIT
TR = TRACE AMOUNT DETECTED

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TABLE II UPPER OTTAWA PAH STUDY

CONC(ppm)

COMPOUND	EXTRACT BLANK	SAMPLE #6	SAMPLE #7	SAMPLE #8	SAMPLE #9	SAMPLE #10	FOUNDERY SAND	MDL (PPM)
BUTYL BENZENE	-			-	-	-	-	0.2
NAPHTHALENE	0.14	0.48	1.10	TR	0.80	0.47	0.11	0.1
2-METHYL NAPHTHALENE	-	0.71	0.29	TR	3.27	1.40	0.11	0.1
DIMETHYL NAPHTHALENE	TR	0.69	TR	-	6.40	0.60	TR	0.2
TRIMETHYL NAPHTHALENE	TR	1.01	0.39	TR	-	7.28	TR	0.1
ACENAPHTHYLENE	-		0.40	-	5.80	TR	-	0.1
ACENAPHTHENE	-	-	0.42	-	1.00	TR	-	0.1
FLUORENE	-	0.13	0.87	-	1.40	-	TR	0.2
PHENANTHRENE	-	1.94	13.26	0.34	5.4	2.13	0.88	0.2
METHYL PHENANTHRENE	TR	6.79	1.70	0.37	-	5.72	-	0.2
ANTHRACENE	-		-	-	1.60	-		0.2
METHYL ANTHRACENE	-		-	-	0.90		-	0.4
FLUORANTHENE	-	0.93	13.26	TR	0.80	4.78	-	0.4
PYRENE	-	0.74	24.19	TR	1.06	4.78		0.4
BENZO(A)ANTHRACENE	-	-	-	TR	1.08	-	-	0.4
CHRYSENE	TR	0.18	24.25	TR	-	1.96	-	0.4
BENZO(A)PYRENE	-	-	-	-	-	-	-	0.6
BENZO(E)PYRENE	-	-	-	-	0.84	-	-	0.6
BENZO(B)FLUORANTHENE	-	-	1.98	-	0.80	2.13	-	0.6
BENZO(K)FLUORANTHENE	-	-	-	-	-	-	-	0.6
PERYLENE	-		-		0.92	-	-	0.5
BENZO(GHI)PERYLENE	-		-		-		-	1.0
INDENO(123-CD)PYRENE	-	TR	8.03		1.46	-	-	1.0
DIBENZO(AH)ANTHRACENE	-	TR	3.17	-	1.50	-	-	1.0
CORONENE	-	-	TR	-		-	-	2.0
1H-PHENALENE	-	-	-	-	-	-	-	5.0
4H-CYCLOPENTA-D,E,F,PHENANTHRENE	-	-	-	-	-	-	-	5.0

- NONE DETECTED
MDL = MINIMUM DETECTION LIMIT
TR = TRACE AMOUNT DETECTED

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TABLE III

UPPER OTTAWA LANDFILL SOIL SAMPLES

PCB ANALYSIS

	PCB Concentrtation (ppm)	PCB Type	MDL (ppm)
1	0.06	Arochlor 1260	0.05
2	0.10	Arochlor 1260	0.05
3	0.28	Arochlor 1242/1260	0.05
4	0.57	Arochlor 1242/1260	0.05
5	0.54	Arochlor 1242/1260	0.05
Blank	N/D		

3.0 ANALYTICAL METHODOLOGY

The PAH analysis was performed using GC/MS (gas chromatography/mass spectrometry) while the PCB's were analysed by ECD/GC (electron capture detection/gas chromatography). Specific details of the analytical methodology are presented in Section 4.0.

All samples were fortified at the 1 ppm level with a mixture containing 8 isotopically labelled PAH standards prior to extraction (references US EPA method #1625). This provides a very accurate means of compound quantification and insures a high level of QA/QC on a per sample basis.

Table IV shows an averaged recovery of the labelled internal standards from the sample matrix.

TABLE IV

LIST OF DEUTERATED PAH INTERNAL STANDARDS
& AVERAGED PERCENT RECOVERY FROM SAMPLE MATRIX

No.	Compound	Natural Quant Ion	Deuterated Quant Ion	% Recovery
1	D_8 Naphthalene	128	136	79
2	D_{10} Acenapthene	154	164	83
3	D_{10} Fluorene	166	176	84
4	D_{10} Anthracene	178	188	92
5	D_{10} Pyrene	202	212	90
6	D_{12} Chrysene	228	240	94
7	D_{12} Benzo(A)Anthracene	228	240	89

3.1 Compound Quantitation

Polynuclear Aromatic Compounds

Quantitation of naturally occurring PAH's was achieved by comparing the peak heights of the natural compound to that of its analogous labelled standard which had been spiked into the sample at a known level. For those target compounds whose corresponding labelled standard was not available, the labelled standard that eluted closest to it was used for quantitation.

3.2 PCB Quantitation

Standard solutions containing 50 ppb (500 pg) of Arochlor 1254 are analysed to insure that a sufficiently low instrument background was present. Background interference was characterized by analysing both neat hexane blanks and solvent extraction blanks.

The sample extracts were analysed and approximate concentrations estimated. Standard solutions at these concentrations were then analysed. This process was repeated until a standard solution that closely resembles the sample both in PCB type and concentration was obtained.

Quantitation of samples containing PCB's is achieved by summing the heights of a minimum of 6 represeneative individual PCB congener peaks in the sample and determining the concentration by comparison to the corresponding peaks in the matched standard of known concentration.

3.3 QA/QC Protocol

a) The use of deuterated internal standards to monitor extraction efficiency assured a confidence level of results on all sample extractions.

b) An extraction blank was analysed to determine the presence of any background interference. These results are presented in Table II.

c) Prior to GC/MS analysis an additional deuterated PAH (D_{10}-Phenanthrene) is added as an internal standard to monitor instrument performance.

d) GC/MS Analysis

Prior to sample analysis reference standards at the 20 ppb level were analysed to demonstrate that the instrument was capable of the required sensitivity. Solvent blanks were run between standards and samples to monitor any possible cross contamination. Reference standards at various levels were analysed after every tenth sample.

Sediment Sample - Extraction Procedure

PAH Analysis

All glassware was detergent washed, rinsed successfully with distilled water, glass distilled acetone and then oven dried; it was then rinsed with Dichloromethane prior to use.

- the entire sample was homogenized and a 10 gr (wet weight, later corrected for % moisture) portion taken and thoroughly mixed with 10 gr of NA_2SO_4
- a mixture of deuterated internal standards (equivalent to 1 ppm) (10 ug) was added to the sample mixture, stirred thoroughly and allowed to equilibrate for 1 hour
- the mixture was extracted with 2 x 50 ml of 10% Aceton e/40% CH_2Cl_2/50% Hexane followed by 2 x 50 ml of acidified ether (1% conc HCl) using sonification and a wrist action shaker (15 min. periods)
- the combined ether fractions were back washed with 2 x 5 ml water to remove the hydrochloric acid
- the organic extracts were combined and dried through Na_2SO_4, 10 ml of isooctane was added and the extract concentrated to 10 mls by rotary evaporation (water bath = 25°C)
- GC/MS analysis for the Base/Neutral fraction was performed directly on the 10 ml extract

Gas Chromatographic/Mass Spectrometry Instrumentation

One ul aliquots of the 10 ml extracts were analysed for the presence of the selected priority pollutants using a Finnigan 4500 GC/MS system set to the following condition:

Mass Spectrometer

Electron Multiplier	-	1450 e.v.
Ionization Energy	-	70 e.v.
Sensitivity	-	10^8 amps/volt
Scan Rate	-	2.25 mSec/mass unit
Scan Range	-	1-2000
Mass Range	-	34-450 a.m.u.

Varian 3500 Gas Chromatograph Conditions

GC Column	-	DB-1 30 M capillary
GC Temp Program		
Initial	-	50°C (0.1 min)
Final	-	285°C (20 min)
Rate	-	-10°C/min
Injection Port Temp	-	250oC
Column Carrier Gas	-	Helium

Instrument and Operating Conditions of Capillary Column ECD/GC

Instrument:	Tracor 460
Columns:	10' x 1/8" S.S. 3% OV-101 on Chromasorb Q
Column Temp:	195° to 250° by 10oC/min
Injection Temp:	275°C
Detection Temp:	330°C
Detection Type:	Electron Capture
Injection Volume:	2 ul
Chart Speed:	1 cm/min

METHOD FOR EXTRACTION OF PCB'S FROM SOIL

- 10 gr soil (corrected for % moisture) or swab sample
- 2 gr Na_2SO_4, is added and the mixture extracted by shaking with 30 ml 20% acetone hexane for 30 min.
- the extract is decanted & filtered through a Na_2SO_4 column
- re-extracted with an additional 30 ml of solvent, ultra-sonic bath 1 min., shaking 5 min.
- add 5 ml isooctane to dried extract
- rot evap. to 2 ml
- apply extract to a column consisting oq 5 gr florosil elute with 50 ml hexane
- concentrate to 1 ml

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